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IS 3904 (1966): Thiometon Concentrates [FAD 1: Pesticides and Pesticides Residue Analysis]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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IS : 3904 - 1966

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2002

Indian Standard

**SPECIFICATION FOR
THIOMETON CONCENTRATES**

(First Reprint JANUARY 2000)

UDC 632.95 : 547.436 THIOMETON

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 5

March 1967

Indian Standard

SPECIFICATION FOR THIOMETON CONCENTRATES

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AMENDMENT NO. 1 SEPTEMBER 1969
TO
IS : 3904-1966 SPECIFICATION FOR THIOMETON
CONCENTRATES

Addenda

(*Page 7, clause A-2.11*) — Add the following new clause after A-2.11:
'A-2.12 Methyl Orange Indicator Solution — 0.1 percent. '

(*Page 7, clause A-3.1*) Add the following at the end of the clause:
'To the filtrate and washings add a drop of methyl orange indicator solution and neutralize the excess acid with concentrated ammonium hydroxide solution. '

Indian Standard

SPECIFICATION FOR THIOMETON CONCENTRATES

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 30 November 1966, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Thiometon concentrates are employed in the preparation of the formulations which are used in the control of pests of agricultural crops.

0.3 Thiometon is a very unstable compound and because of this property, the concentration of active ingredient in this compound as obtained from manufacture is not less than 50 percent and, therefore, is stabilized. The concentration of active ingredient in the technically pure thiometon could be as high as 95 percent and this high concentration is obtained by distillation in the laboratory. It is kept refrigerated in order to keep up the concentration at that level. This product is used for analytical purposes. In view of the above, minimum percentage of the active ingredient in the compound has been fixed as 50 and is taken as commercially pure product.

0.4 This standard is one of a series of Indian Standards on pesticides and their formulations.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of test for thiometon concentrates employed in the preparation of formulations used in the control of the pests of agricultural crops.

2. REQUIREMENTS

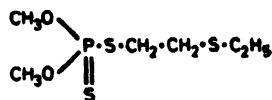
2.1 Description — The material shall be in form of a clear, blue liquid, free from extraneous impurities.

*Rules for rounding off numerical values (*revised*).

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2.2 Chemical Name — O,O-dimethyl-S-[2-(ethylthio) ethyl] phosphorodithionate.

2.3 Structural Formula



2.4 Empirical Formula — $\text{C}_8\text{H}_{18}\text{O}_2\text{S}_3\text{P}$.

2.5 Molecular Weight — 246.26.

2.6 The material shall comply with the requirements as specified in Table 1.

TABLE 1 REQUIREMENTS FOR THIOMETON CONCENTRATES

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO APPENDIX)
(1)	(2)	(3)	(4)
i)	O,O-dimethyl-S-[2-(ethylthio) ethyl] phosphorodithionate content, percent by weight, <i>Min</i>	50.0	A
ii)	Water content, percent by weight, <i>Max</i>	0.1	B
iii)	Acidity (as H_2SO_4), percent by weight, <i>Max</i>	0.04	C
iv)	Material insoluble in acetone, percent by weight, <i>Max</i>	0.01	D
v)	Specific gravity at 25°/25°C	1.035 to 1.045	E

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in suitable, clean and dry containers made of steel or any other suitable containers as agreed to between the purchaser and the vendor.

3.2 Marking — The container shall be securely closed and shall bear legibly and indelibly the following information:

- Name of the material,
- Name of the manufacturer,
- Date of manufacture,
- Batch number,
- Net volume of contents, and

f) The minimum cautionary notice worded as under:

'THIOMETON IS HIGHLY TOXIC. AVOID CONTAMINATION OF FOODSTUFFS AND ANIMAL FEEDS, AND INHALATION OF DUSTS AND MISTS MADE FROM THIS INSECTICIDE. IF IT COMES IN CONTACT WITH SKIN, WASH WITH SOAP AND WATER. DO NOT USE THIS CONTAINER FOR ANY OTHER PURPOSE EXCEPT FOR STORAGE OF PESTICIDES. IN CASE OF POISONING CALL A PHYSICIAN. ANTIDOTE — ATROPINE SUPPORTED BY 2 PAM (2 PYRIDINE-2-ALDOXINE-N-METHYL-IODIDE).'

3.2.1 In addition to the above, the container shall also be marked with the symbol for danger of poisoning as specified in IS : 1260-1958*

3.2.2 The containers may also be marked with the Standard Mark.

3.2.3 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The details of conditions under which the licence for the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in Appendix F.

5. TESTS

5.1 Tests shall be carried out as prescribed in the appropriate appendices as specified in col 4 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960†) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Code of symbols for labelling of dangerous goods.

†Specification for water, distilled quality (*revised*).

APPENDIX A

[Table 1, Item (i)]

DETERMINATION OF O,O-DIMETHYL-S- [2-(ETHYLTHIO) ETHYL] PHOSPHORODI- THIONATE

A-1. PRINCIPLE OF METHOD

A-1.1 Thiometon is decomposed by heating it with hydrogen peroxide and concentrated sulphuric acid, and the phosphate obtained estimated either gravimetrically or volumetrically by the ammonium molybdate method.

A-2. REAGENTS

A-2.1 Hydrogen Peroxide — 30 percent (*w/w*).

A-2.2 Concentrated Sulphuric Acid — conforming to IS : 266-1961*.

A-2.3 Concentrated Nitric Acid — sp gr 1.42 (conforming to IS : 264-1950†).

A-2.4 Ammonium Molybdate Reagent — Dissolve 100 g of molybdic anhydride or 118 g of molybdic acid in a mixture of 400 ml of water and 80 ml of concentrated ammonia. Filter, if necessary. Add this solution slowly and with constant stirring into a solution containing 400 ml of concentrated nitric acid and 600 ml of water. Allow the resulting solution to stand for several days in a warm place or until a portion heated to 40° to 45°C gives no yellow precipitate. Preserve the decanted solution in a glass-stoppered bottle.

A-2.5 Wash Liquid — Dissolve 10 g of ammonium nitrate in water, add 8 ml of concentrated nitric acid and dilute with water to 200 ml.

A-2.6 Potassium Ferrocyanide Solution — 5 percent (*w/v*).

A-2.7 Magnesia Mixture — Dissolve 55 g of crystallised magnesium chloride ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$) in water, and after adding 140 g of ammonium chloride and 130 ml of ammonia, dilute to one litre.

A-2.8 Ammonium Hydroxide Solution for Washing — 2.5 percent (*w/v, min*).

A-2.9 Standard Sodium Hydroxide Solution — 0.5 N, carbonate free.

A-2.10 Standard Hydrochloric Acid — 0.5 N.

*Specification for sulphuric acid (revised).

†Specification for nitric acid.

A-2.11 Phenolphthalein Indicator Solution — Dissolve 0.1 g of phenolphthalein in 60 ml of rectified spirit conforming to IS : 323-1959* and dilute with distilled water to 100 ml.

A-3. PROCEDURE

A-3.1 Digestion — Weigh 0.2 to 0.4 g of thiometon concentrates in a glass-ampoule and introduce it in a 500-ml Kjeldahl digestion flask. Add 20 ml of hydrogen peroxide and 10 ml of concentrated sulphuric acid and heat over a microburner until white fumes of sulphur trioxide come out. Allow to cool, add 10 ml of concentrated nitric acid and heat again to fumes of sulphur trioxide. Cool, add 20 ml of hydrogen peroxide and heat again till white fumes are given off. After cooling add 100 ml of water, and evaporate on a water-bath to 30 to 40 ml. Filter through a filter paper and wash with water till washings are neutral.

A-3.2 Gravimetric Method

A-3.2.1 Acidulate solution obtained in A-3.1 with nitric acid, heat to 80°C, add excess of ammonium molybdate reagent (at the rate of about 75 ml reagent for every 50 mg of P) and digest at 65°C for one hour. Test for complete precipitation by adding more molybdate reagent to the clear supernatant.

A-3.2.2 Filter precipitate and wash with the wash liquid till only a faint brown colour is given on testing 5 ml of the washings with a little potassium ferrocyanide solution.

A-3.2.3 Dissolve the precipitate on the filter paper with the ammonium hydroxide solution (1 + 1) and hot water and wash into a beaker to volume not more than 100 ml. Neutralize with hydrochloric acid (1 + 1) to litmus paper or bromothymol blue indicator. Cool, and add slowly and with vigorous stirring magnesia mixture at the rate of about 20 ml per every 50 mg of P present. After 15 minutes add 12 ml of concentrated ammonia, let stand until supernatant is clear (usually two hours but preferably kept overnight), filter and wash with ammonium hydroxide solution till the washings are free from chloride ions. Dry, burn at low heat, ignite to constant weight at 950° to 1 000°C, cool in a desiccator and weigh as $Mg_2P_2O_7$. Convert to P by multiplying the weight obtained by 0.278 3.

A-3.3 Volumetric Method

A-3.3.1 Treat the ammonium phosphomolybdate precipitate obtained in A-3.2.2 along with the filter paper with a slight excess of standard sodium hydroxide solution (say, 50 ml). Stopper the flask and shake till the precipitate dissolves completely. Dilute to 150 ml with water and titrate with 0.5 N hydrochloric acid using phenolphthalein indicator until the pink

*Specification for rectified spirit (revised).

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colour is completely discharged. Complete titration with 0.5 N alkali to the reappearance of the pink colour.

1 ml 0.5 N NaOH = 0.000 674 5 g P

A-4. CALCULATION

A-4.1 O,O-dimethyl-S-[2-(ethylthio) ethyl] phosphorodithionate content, percent by weight = $\frac{w_1 \times 100 \times 100}{w \times 12.6}$

where

w_1 = weight in g of P obtained as in A-3.2.3 or A-3.3.1, and

w = weight in g of sample taken for the test.

A P P E N D I X B

[Table 1, Item (ii)]

DETERMINATION OF WATER CONTENT

B-1. PRINCIPLE OF METHOD

B-1.1 The method is based on the reaction of water with iodine and sulphur dioxide in pyridine/methyl alcohol solution (Karl Fischer reagent). The pyridine serves to prevent loss of sulphur dioxide from the reagent by uniting with it to form an additive compound, and also facilitates completion of the reaction with water by combining with the reaction product.

B-2. APPARATUS

B-2.1 The apparatus required, a suitable arrangement of which is shown in Fig. 1, consists of the following parts.

B-2.1.1 Reaction Vessel — Of working capacity of not less than 60 ml. The wide neck terminates in a suitable air-tight detachable junction, fitted to accommodate a burette jet (extended to suitable length) and tubes for entry and exit of nitrogen, the inlet tube extending nearly to the bottom of the vessel. The nitrogen supply is passed through a drying-train consisting of a guard-tube filled with freshly activated silica-gel or other efficient desiccant, and a flask containing Karl Fischer reagent. Two platinum-wire electrodes extending nearly to the bottom of the reaction vessel are either fused in through the wall of the vessel or inserted through the seal, if preferred. A side arm, closed by a stopper or a vaccine cap, provides for insertion of the sample.

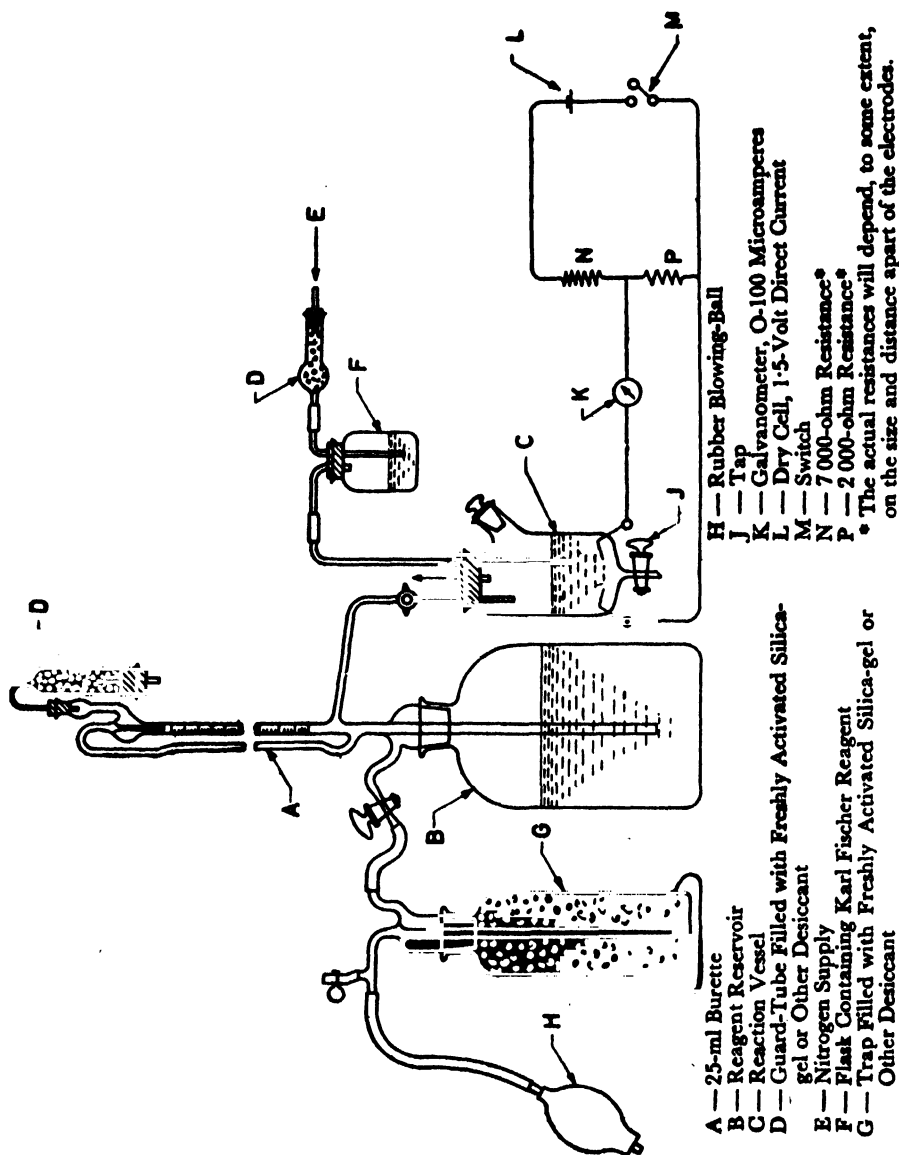


FIG. 1 APPARATUS FOR THE DETERMINATION OF WATER CONTENT

B-2.1.2 Burette — 25 ml capacity, with pressure-filling device and automatic zero, fitted with a guard-tube filled with freshly activated silica-gel or other efficient desiccant. The reagent reservoir forming part of the pressure-filling device is protected by a trap filled with freshly activated silica-gel or other efficient desiccant.

B-2.1.3 Electrical Circuit — incorporating a dry cell or other source of 1.5 volt direct current, a potential divider to provide a working potential of about 300 mV, a galvanometer with a sensitivity such that full-scale deflection is obtained when a current of not more than 100 μ A is passed, and a switch. The galvanometer may be in series with the reaction vessel or in a shunt position. In the former case the galvanometer will give a 'null' reading at the end-point, and in the latter case a 'positive' reading.

B-2.1.4 Bulb Pipettes — one-mark, of capacity 5 ml, 10 ml, and 20 ml, with a cotton-wool plug in the upper part above the calibration mark.

B-3. REAGENTS

B-3.1 Anhydrous Methyl Alcohol — If not already anhydrous, dry by distillation over magnesium turnings and a little iodine, or by fractionation through a 1.5-m rectifying column, and distil into a receiver protected from atmospheric moisture by a guard-tube. Methyl alcohol dried in this manner will contain not more than 0.03 percent of water.

B-3.2 Standard Solution of Water in Methyl Alcohol — Dry thoroughly in an oven a 100-ml graduated flask and allow to cool in a desiccator. Partially fill the flask with the anhydrous methyl alcohol, add about 0.5 g of distilled water, accurately weighed, from a weighing pipette, make up to the mark with the methyl alcohol, and mix thoroughly by shaking. This solution should be prepared immediately before use.

B-3.3 Pyridine — Distil the pyridine, collecting the fraction boiling between 114° and 116°C at 760 mmHg pressure, the receiver being protected from atmospheric moisture by a guard-tube.

B-3.4 Nitrogen, Dry — This should be supplied from a cylinder under pressure.

B-3.5 Karl Fischer Reagent — This reagent should have a water equivalent of between 3.0 and 4.0 mg/ml and may be prepared as follows.

B-3.5.1 Measure 800 ml of the anhydrous methyl alcohol into a dry 1 500-ml glass-stoppered flask and add 160 g of pyridine. Exchange the glass stopper for a two-hole rubber bung fitted with an inlet tube reaching nearly to the bottom of the flask and a short outlet tube protected by a guard-tube. Weigh the flask with its fittings and contents and cool in ice water. Connect the inlet tube to a glass siphon containing sulphur dioxide of refrigeration grade, and allow the gas to pass into the mixture

with continuous agitation, until the total weight of the flask and its contents is increased by about 40 g. Then remove the rubber bung and delivery tubes and add 90 g of resublimed iodine. Replace the glass stopper, shake to dissolve the iodine, and allow the mixture to stand for 24 hours. Finally, transfer to the reagent reservoir. The water equivalent of the Karl Fischer reagent thus prepared will be about 3.5 mg/ml.

B-4. PROCEDURE

B-4.1 Standardization of the Karl Fischer Reagent

B-4.1.1 Transfer 10 ml of the anhydrous methyl alcohol to the reaction vessel, through the side arm, by means of a pipette. Turn on a slow stream of nitrogen, sufficient to produce adequate agitation, close the electrical circuit and titrate with the Karl Fischer reagent until a large deflection of the galvanometer is obtained and remains above the half-scale reading for at least 30 seconds. Ignore the volumes or the reagents used up to this stage. Run out most of the reaction mixture, leaving just enough to cover the drain tap. (This operation is intended to remove any moisture in the reaction vessel.)

B-4.1.2 Transfer to the reaction vessel a further 10 ml of the methyl alcohol and titrate as in B-4.1.1 with the Karl Fischer reagent, noting the volume used. Then add to the reaction vessel 10 ml of the standard solution of water in methyl alcohol and again titrate with the Karl Fischer reagent.

B-4.1.3 Calculate the water equivalent of the Karl Fischer reagent as follows:

$$\text{Water equivalent of the Karl Fischer reagent (mg/ml)} = \frac{100 \times W}{V_2 - V_1}$$

where

W = weight in g of water used in preparing 100 ml of the standard solution of water in methyl alcohol,

V_2 = volume in ml of the Karl Fischer reagent used for titration of the standard solution of water in methyl alcohol, and

V_1 = volume in ml of the Karl Fischer reagent used for titration of the methyl alcohol alone.

B-4.2 Analysis of the Material

B-4.2.1 Add to the reaction vessel 10 ml of the anhydrous methyl alcohol. Turn on a slow stream of nitrogen, sufficient to produce adequate agitation, switch on the current, and titrate with the Karl Fischer reagent as

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described in B-4.1.1. Ignore this titration. Immediately add an amount of the sample containing between 0.06 and 0.08 g of water (sufficient to give a titration of about 20 ml of the reagent) and titrate with the Karl Fischer reagent.

B-4.2.2 Carry out a blank titration on a volume of the anhydrous methyl alcohol equal to that present in the aliquot of the sample solution.

B-5. CALCULATION

B-5.1 Water content, percent by weight = $\frac{F \times V}{v \times S \times 10}$

where

F = water equivalent of the Karl Fischer reagent (mg/ml)
(see B-4.1.3),

V = volume in ml of the Karl Fischer reagent used,

v = volume in ml of the material taken for the test, and

S = specific gravity of the material at room temperature*.

APPENDIX C

[Table 1, Item (iii)]

DETERMINATION OF ACIDITY

C-1. REAGENTS

C-1.1 Phenolphthalein Indicator Solution — Dissolve 0.1 g of phenolphthalein in 100 ml of rectified spirit (conforming to IS : 323-1959†).

C-1.2 Standard Sodium Hydroxide Solution — 0.02 N.

C-2. PROCEDURE

C-2.1 Weigh accurately about 5 g of the material and shake it with exactly 50 ml of water. Allow to settle, filter by decantation and titrate 25 ml of the filtrate with the standard sodium hydroxide solution using phenolphthalein as the indicator.

C-2.2 Carry out a blank determination using 25 ml of water.

*For the purpose of this calculation the specific gravity of the material at 15.6°C, 20°C or 25°C may be used.

†Specification for rectified spirit (revised).

D-3. CALCULATION

D-3.1 Acidity (as H_2SO_4), percent by weight
$$= \frac{4.9 (V - v) N}{W}$$

where

V = volume in ml of the standard sodium hydroxide solution required for the test with the material,

v = volume in ml of the standard sodium hydroxide required for the blank determination,

N = normality of the standard sodium hydroxide solution, and

W = weight in g of the material taken for the test.

A P P E N D I X D

[*Table 1, Item (iv)*]

**DETERMINATION OF MATERIAL INSOLUBLE IN
ACETONE**

D-1. REAGENT**D-1.1 Acetone****D-2. PROCEDURE**

D-2.1 Weigh 10 g of the material into a clean and dry 250-ml Erlenmeyer flask fitted with a ground-glass joint. Add 150 ml of dry acetone and warm under reflux until all soluble material is dissolved. Filter the solution using a tared Gooch or sintered-glass crucible of porosity No. 3, and wash well with more solvent. Dry at 110°C for 30 minutes, cool and weigh.

D-3. CALCULATION

D-3.1 Matter insoluble in acetone, percent by weight
$$= \frac{100 w}{W}$$

where

w = weight in g of the residue obtained after drying, and

W = weight in g of the material taken for the test.

APPENDIX E

[Table 1, Item (v)]

DETERMINATION OF SPECIFIC GRAVITY

E-1. TERMINOLOGY

E-1.1 For the purpose of this standard, the specific gravity of the material shall be the ratio of the weight in air of a given volume of the material at 25°C to that of an equal volume of water at the same temperature.

E-2. METHOD

E-2.1 The specific gravity shall be determined with a specific gravity bottle — Castell Evans modification with ground-in stopper and ground-on cap — (*see* Fig. 2) or with a pyknometer tube of the Perkin's type (*see* Fig. 3).

E-3. PROCEDURE

E-3.1 Weigh the clean and dry specific gravity bottle or pyknometer of 25 ml capacity. Fill the tared specific gravity bottle or pyknometer with freshly boiled water and place it in a bath maintained at $25^{\circ} \pm 1^{\circ}\text{C}$ and allow sufficient time to attain the temperature of the bath. Then insert the ground-in stopper which has also been brought to $25^{\circ} \pm 1^{\circ}\text{C}$. Wipe the excess liquid from the stopper and place the cap in position on it. Remove the specific gravity bottle or the pyknometer from the bath, bring to room temperature and weigh. Empty the specific gravity bottle or the pyknometer, clean and dry it, and repeat the operation with the material at $25^{\circ} \pm 1^{\circ}\text{C}$.

E-4. CALCULATION AND REPORT

E-4.1 Specify the temperature of testing in the report.

E-4.2 The specific gravity is calculated by the following formula:

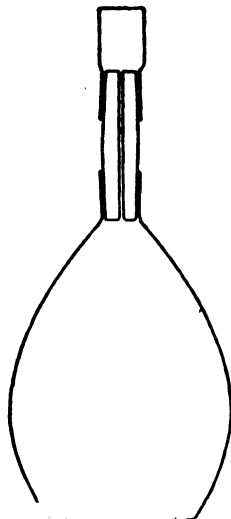
$$\text{Specific gravity at } 25^{\circ}/25^{\circ}\text{C} = \frac{C - A}{B - A}$$

where

C = weight in g of the specific gravity bottle or pyknometer filled with the material at 25°C,

A = weight in g of the dry specific gravity bottle or pyknometer, and

B = weight in g of the specific gravity bottle or pyknometer filled with water at 25°C.



**FIG. 2 SPECIFIC GRAVITY BOTTLE (CASTELL EVANS MODIFICATION
WITH GROUND-IN STOPPER AND GROUND-ON CAP)**

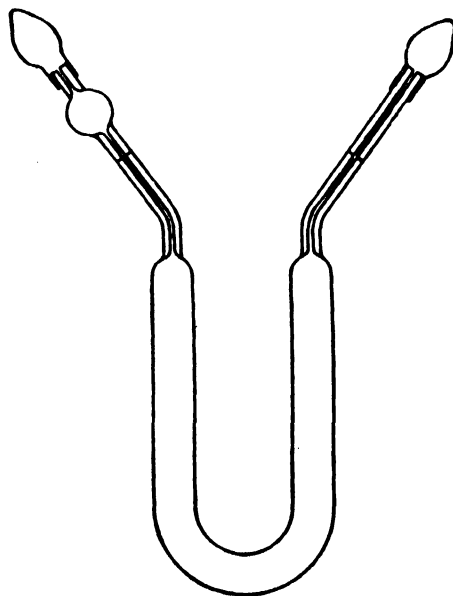


FIG. 3 PYCNOMETER TUBE (PERKIN'S TYPE)

APPENDIX F

(Clause 4.1)

SAMPLING OF THIOMETON CONCENTRATES

F-1. GENERAL PRECAUTIONS

F-1.0 In drawing, preparing, storing, and handling test-samples, the following precautions and directions shall be observed.

F-1.1 Samples shall not be taken in an exposed place.

F-1.2 The sampling instrument shall be of glass and it shall be clean and dry when used.

F-1.3 Proper precautions shall be taken while drawing samples since the material is highly poisonous.

F-1.4 Precautions shall be taken to protect the samples, the material being sampled, the sampling instruments, and the containers for samples from adventitious contamination.

F-1.5 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by shaking or stirring or both, by suitable means or by rolling so as to bring all portions into uniform distribution.

F-1.6 The samples shall be placed in leak-proof 100-ml aluminium containers.

F-1.7 The sample containers shall be of such a size that they are almost, but not completely, filled by the sample.

F-1.8 Each sample container shall be fitted with an inner stopper, which shall be sealed on the neck of the container by means of a suitable sealing composition. The container shall then be securely closed by screw cap aluminium closures. The containers shall then be marked with full details of sampling, the date of manufacture, name of the manufacturer, and other particulars of the consignment.

F-1.9 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

F-1.10 Samples shall be protected from light.

F-2. SAMPLING INSTRUMENT

F-2.1 Sampling Tube — It shall be made of thick glass of 20 to 40 mm diameter and 400 to 800 mm in length (see Fig. 4). The upper and lower

ends are conical and of 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For taking a sample, the tube is first closed at the top with the thumb or a stopper and lowered until the desired depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn.

F-2.1.1 For small containers, a smaller sampling tube of suitable dimensions may be used.

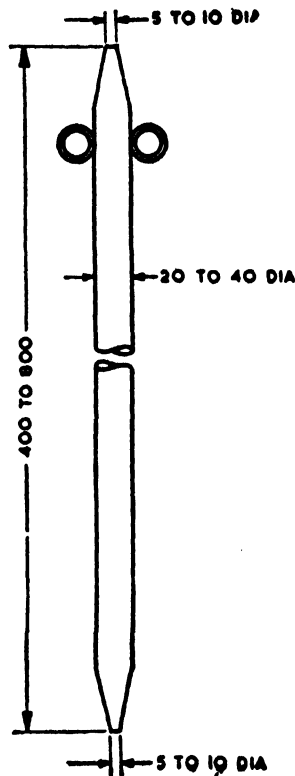


FIG. 4 SAMPLING TUBE

F-3. SCALE OF SAMPLING

F-3.1 Lot — All the containers in a single consignment of the material drawn from the same batch of manufacture shall constitute a lot. If a consignment is declared or is known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

F-3.1.1 Samples shall be tested separately for each lot for ascertaining the conformity of the material to the requirements of this specification.

F-3.2 The number n of containers to be selected from the lot shall depend on the size of the lot N and shall be in accordance with col 1 and 2 of Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

Lot Size	No. of Containers to be Selected
N	n
(1)	(2)
Up to 15	3
16 „ 40	4
41 „ 65	5
66 „ 110	7
Over 110	10

F-3.3 These containers shall be selected at random from the lot. To ensure the randomness of selection, a random number table as agreed to between the purchaser and the supplier shall be used. In case such a table is not available, the following procedure shall be adopted:

Starting from any container in the lot, count them as 1,2,3,....., up to r in one order, where r is equal to the integral part of the value of N/n ; N being the total number of containers in the lot and n the number of containers to be selected (see Table 2). Every r th container thus counted shall be separated until the requisite number of containers is obtained from the lot to give the sample for test.

F-4. TEST SAMPLES AND REFEREE SAMPLES

F-4.1 Before drawing the test samples, mix thoroughly the contents of each container selected, by shaking or stirring by suitable means or by rolling. Draw, by inserting the sampling instrument through the bung hole or any other convenient opening, small portions of the material from different parts of each selected container. The total quantity of the material drawn from each container shall be not less than 350 ml.

F-4.2 Mix thoroughly all portions of the material drawn from the same container. Out of these portions, a small but equal quantity shall be taken from each selected container and shall be well mixed together so as to form a composite sample of not less than 150 ml. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and third for the referee.

F-4.3 The remaining portions of the material from each container (after the required quantity needed for formation of the composite sample has been taken out) shall be divided into three equal parts. These parts shall be immediately transferred to thoroughly dried receptacles which are then sealed air-tight and labelled with all the particulars of sampling given under **F-1.8**. The material in each such sealed receptacle shall constitute a test sample. These individual samples shall be separated into three identical sets of test samples in such a way that each set has a sample representing each selected container one of these three sets shall be marked for the purchaser, another for the supplier, and the third for the referee.

F-4.4 Referee Samples — Referee samples shall consist of the composite sample (*see* **F-4.2**) and a set of individual test samples (*see* **F-4.3**) marked for this purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the two.

F-5. TESTING OF SAMPLES

F-5.0 The number of tests mentioned in **F-5.1** and **F-5.2** shall be first conducted on the test samples marked for the purchaser. In the contingencies (such as loss, spoilage, spilling of the purchaser's test samples) or if the supplier so desires, the test samples marked for the supplier may be tested. In case of a dispute, the referee test sample shall be tested and the test results obtained on the referee test samples shall be considered as final.

F-5.1 Tests for description (*see* **2.1**) and the determination of thiometon content shall be conducted individually on each of the samples constituting a set of test samples (*see* **F-4.3**).

F-5.2 Tests for the determination of the remaining characteristics, namely, water content, material insoluble in acetone, specific gravity, and acidity shall be conducted on the composite sample as prepared under **F-4.2**.

F-6. CRITERIA FOR CONFORMITY

F-6.1 A lot shall be declared as conforming to the specification, when:

- a) each of the individual samples satisfies the requirement given in **2.1**;
- b) each of the test results for thiometon content satisfies the corresponding requirement specified in Table 1. If, however, one or more test results do not satisfy the specified requirements, the conformity of the lot shall be ascertained in accordance with **F-6.1.1**; and
- c) the test results on the composite sample for water content, material insoluble in acetone, specific gravity, and acidity, satisfy the corresponding requirements given in Table 1.

F-6.1.1 In case one or more of the test results do not satisfy the requirements for thiometon content, the following procedure shall be adopted for determining conformity of the material in respect of this characteristic. The mean and the range of the corresponding test results shall be calculated as follows:

$$\text{Mean } (\bar{X}) = \frac{\text{Sum of the test results}}{\text{Number of the test results}}$$

$$\text{Range } (R) = \text{Difference between the highest and the lowest values obtained for the test results}$$

The appropriate expression as shown in col 5 of Table 3 shall be calculated. If the value of this expression satisfies the relevant condition as given in col 5 of Table 3, the lot shall be declared to have satisfied the requirement for thiometon content.

TABLE 3 CRITERION FOR CONFORMITY

CHARACTERISTIC	TEST RESULTS 1, 2, 3,, n	AVERAGE	RANGE	CRITERION FOR CONFORMITY
(1)	(2)	(3)	(4)	(5)
Thiometon content, percent by weight	—	\bar{X}	R	$(\bar{X} - 0.6 R) \geq 50.0$

(Continued from page 2)

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